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Preparation and characterization of activated carbon from brown algae (*Sargassum sp.*) using phosphoric acid activation for heavy metal adsorption

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Abstract

This study presents the successful preparation and evaluation of activated carbon derived from brown seaweed (Sargassum sp.) waste using chemical activation with zinc chloride ($ZnCl_2$) and thermal treatment. The resulting biosorbent was characterized by its iodine number, surface morphology, and adsorption efficiency for hexavalent chromium (Cr^{6+}) ions in aqueous solutions. The activated carbons exhibited high microporosity, with iodine numbers reaching 290 mg/g at 800 °C, closely comparable to commercial activated carbon. SEM analysis revealed a porous surface structure, supporting its high adsorption capacity.

Batch adsorption experiments demonstrated that Cr^{6+} removal efficiency was strongly influenced by solution pH, initial ion concentration, and contact time. Optimal removal (>90%) was achieved at pH 2, low metal concentrations, and a contact time of 120 minutes. The bio-based activated carbon showed promising potential as a cost-effective and environmentally friendly material for heavy metal remediation. This approach contributes to sustainable waste valorization and supports circular economy practices.

Keywords: Brown seaweed; Activated carbon; Zinc chloride activation; Cr^{6+} adsorption; Heavy metals; Sustainable water treatment; Microporosity; Bio-based adsorbent

1 Introduction

The increasing contamination of water resources by heavy metals such as lead (Pb^{2+}), cadmium (Cd^{2+}), and mercury (Hg^{2+}) has raised serious environmental and public health concerns globally. Unlike organic pollutants, heavy metals are non-biodegradable, persist in ecosystems, and tend to bioaccumulate in the food chain, leading to severe toxicological effects in humans and animals [1,4]. Conventional remediation technologies such as chemical precipitation, ion exchange, and membrane filtration are often costly, inefficient at low concentrations, and generate secondary waste [6].

Activated carbon (AC) has been widely recognized as a highly effective adsorbent for heavy metal removal due to its high surface area, porosity, and surface functional groups [2]. However, commercial activated carbons are typically derived from non-renewable sources like coal or petroleum-based materials, leading to concerns about sustainability and production cost.

In recent years, there has been growing interest in the development of bio-based activated carbons from agricultural or marine waste, aligning with circular economy principles and sustainable development goals. Among marine biomass resources, brown seaweed (*Sargassum* spp.) is particularly promising due to its abundance in tropical and subtropical regions, high carbon content, and natural composition of polysaccharides such as alginate, fucoidan, and laminarin, which can serve as efficient carbon precursors [3,5].

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This study aims to develop a cost-effective and environmentally friendly activated carbon from Sargassum spp. collected from Vietnamese coastal areas. Using chemical activation with phosphoric acid (H_3PO_4) and subsequent thermal carbonization, the resultant biosorbent was characterized for its physicochemical properties (surface area, pore structure, and functional groups) and evaluated for its efficiency in removing heavy metals (Pb^{2+} and Cd^{2+}) from aqueous solutions. The research not only contributes to sustainable water treatment technologies but also valorizes marine biomass waste, thus promoting both environmental protection and resource recovery.

2 Material and methods

2.1 Preparation of seaweed residue biomass

Brown seaweed waste (*Sargassum* sp.) was collected from the processing site, thoroughly washed first with tap water and then with distilled water to completely remove salts and other impurities. The cleaned biomass was air-dried under ambient conditions, ground to a fine powder, and then sieved to obtain two particle-size fractions: material MA with particle size less than 0.5 mm and material MB with particle size greater than 0.5 mm. Both fractions were placed in airtight containers to prevent moisture uptake and stored for subsequent experiments.

2.2 Chemical activation and carbonization

Twenty grams (20 g) of dried seaweed powder from each fraction was soaked in 200 mL of 20% (w/v) zinc chloride ($ZnCl_2$) solution. The mixture was stirred continuously for 2 hours to ensure uniform impregnation. After soaking, the excess $ZnCl_2$ solution was decanted, and the impregnated biomass was dried in a hot-air oven at 100 ± 2 °C for 24 hours.

The dried, $ZnCl_2$-impregnated biomass was then transferred into ceramic crucibles and subjected to pyrolysis in a muffle furnace under limited air supply. Two carbonization temperatures were tested: 600 °C and 800 °C, with a residence time of 2 hours. The furnace temperature was increased rapidly from room temperature to the target temperature.

2.3 Post-treatment and washing

After carbonization, the activated carbon was allowed to cool and then washed with 0.5 N HCl to remove residual $ZnCl_2$. The sample was further rinsed with warm distilled water until the washings reached neutral pH (~7.0), indicating the complete removal of free Zn^{2+} ions. The cleaned carbon was dried at 105 °C for 12 hours in a hot-air oven. The final product was weighed to determine the carbon yield.

2.4 Determination of Iodine Number

The iodine number, which reflects the microporosity and adsorption capacity of activated carbon, was determined using the standard iodine adsorption method. In this procedure, 0.1 g of activated carbon was accurately weighed into a 200 mL Erlenmeyer flask and mixed with 40 mL of a standard iodine solution of known initial concentration (C_1, mol/L). The mixture was shaken for 15 minutes to ensure complete adsorption equilibrium. After equilibration, the residual iodine in the solution was titrated with 0.05 M sodium thiosulfate ($Na_2S_2O_3$) using starch as an indicator. The iodine number (Q, mg/g) was then calculated from the initial and final iodine concentrations and volumes according to the following relationship:

Q=mAC(C1V1−C2V2)×mI

$$Q = \frac{(C_1V_1 - C_2V_2)m_I}{m_{AC}}$$

where C_1 and C_2 are the initial and equilibrium iodine concentrations (mol/L), V_1 and V_2 are the initial and final volumes (mL), m_I is the molecular weight of iodine (254 g/mol), and m_{AC} is the mass of activated carbon used (g).

2.5 Characterization of Activated Carbon

Surface Morphology: The morphological structure of the activated carbon was observed using Scanning Electron Microscopy (SEM), which revealed the development of porous cavities after activation.

2.6 Data analysis

Data was analysed with descriptive statistics and removed outliers using the Duncan method.

3 Results and discussion

3.1 Iodine number of activated carbon

The iodine number, an important indicator of microporosity and adsorption capacity of activated carbon, was used to evaluate the quality of the prepared bio-based activated carbons. As shown in Table 1, the iodine number varied with activation temperature and type of carbon:

Table 1 Iodine number of activated carbons at different activation temperatures

Type of Activated Carbon	Iodine Number (mg/g)
Activated carbon from seaweed at 800 °C	290
Activated carbon from seaweed at 600 °C	276
Commercial activated carbon (control)	303

Among the three types, commercial activated carbon exhibited the highest iodine number (303 mg/g), indicating its superior microporosity. However, the activated carbons produced from *Sargassum sp.* waste at both 800 °C and 600 °C also showed high iodine values (290 mg/g and 276 mg/g, respectively), demonstrating that seaweed biomass can be effectively converted into porous adsorbents.

Notably, the sample activated at 800 °C achieved a higher iodine number than that at 600 °C, suggesting that elevated activation temperatures enhance the development of microporous structure, thereby improving the adsorption capability. This result is consistent with previous findings where thermal activation at higher temperatures often increases surface area and pore development.

3.2 Effect of pH on Cr^{6+} adsorption capacity

To further evaluate the adsorption performance of the activated carbon, the removal efficiency of hexavalent chromium (Cr^{6+}) from aqueous solution (10 mg/L) was assessed as a function of pH after 60 minutes of contact time (Figure 1).

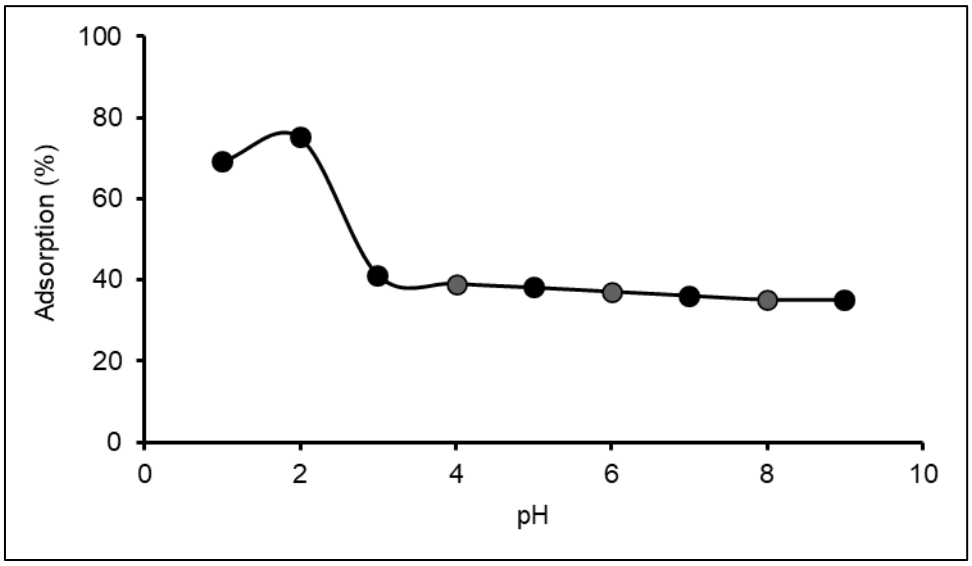

Figure 1 Effect of pH on Cr^{6+} adsorption capacity

As observed, the adsorption efficiency was highly dependent on the solution pH. The removal percentage was highest in acidic conditions, reaching a peak at around pH 2, with an efficiency exceeding 70%. As the pH increased, the adsorption efficiency sharply declined and plateaued at pH 5–9, with values below 40%.

This behavior can be attributed to the speciation of Cr^{6+} and the surface charge of the activated carbon. Under acidic conditions, Cr^{6+} primarily exists as $HCrO_4^-$, which interacts more effectively with positively charged functional groups on the carbon surface. As pH increases, the surface becomes less protonated and Cr^{6+} converts to CrO_4^{2-} or $Cr_2O_7^{2-}$, leading to electrostatic repulsion and reduced adsorption.

These findings suggest that the adsorption of Cr^{6+} onto the bio-based activated carbon is most effective in acidic environments, with pH 2 being optimal for removal.

3.3 Effect of Cr^{6+} concentration on the adsorption efficiency of activated carbon

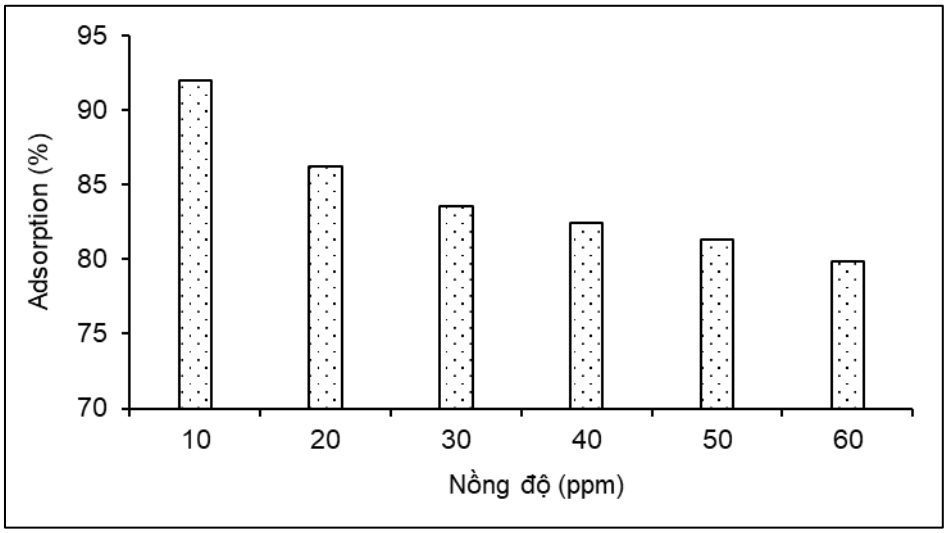

Figure 2 Effect of Cr^{6+} concentration on the adsorption efficiency of activated carbon

The influence of the initial Cr^{6+} concentration on the adsorption efficiency of activated carbon at pH 2, using 0.6 g of adsorbent, was evaluated across a concentration range from 10 to 60 ppm. The results are illustrated in Figure 2. A clear decreasing trend in adsorption efficiency was observed with increasing metal ion concentration.

At the lowest tested concentration (10 ppm), the removal efficiency reached a maximum of approximately 92%, indicating a highly effective interaction between the Cr^{6+} ions and the available adsorption sites on the activated carbon surface. However, as the initial concentration increased, the adsorption efficiency steadily declined, with the lowest efficiency of around 79% recorded at 60 ppm.

This phenomenon can be explained by the saturation of active sites on the surface of the adsorbent. At low concentrations, the number of Cr^{6+} ions is sufficiently small compared to the number of available adsorption sites, facilitating effective adsorption. As the concentration increases, the competition between Cr^{6+} ions for the limited number of active sites intensifies, leading to a gradual reduction in removal efficiency.

Additionally, higher metal ion concentrations may increase the ionic strength of the solution, potentially interfering with the electrostatic interactions between Cr^{6+} ions and the adsorbent surface. Furthermore, at high concentrations, diffusion limitations may arise, reducing the access of Cr^{6+} ions to internal pores of the activated carbon.

These findings are consistent with previous studies, which also reported a negative correlation between initial metal ion concentration and adsorption efficiency when using a fixed amount of adsorbent under constant pH conditions.

3.4 Effect of contact time on the adsorption of Cr^{6+} Ions

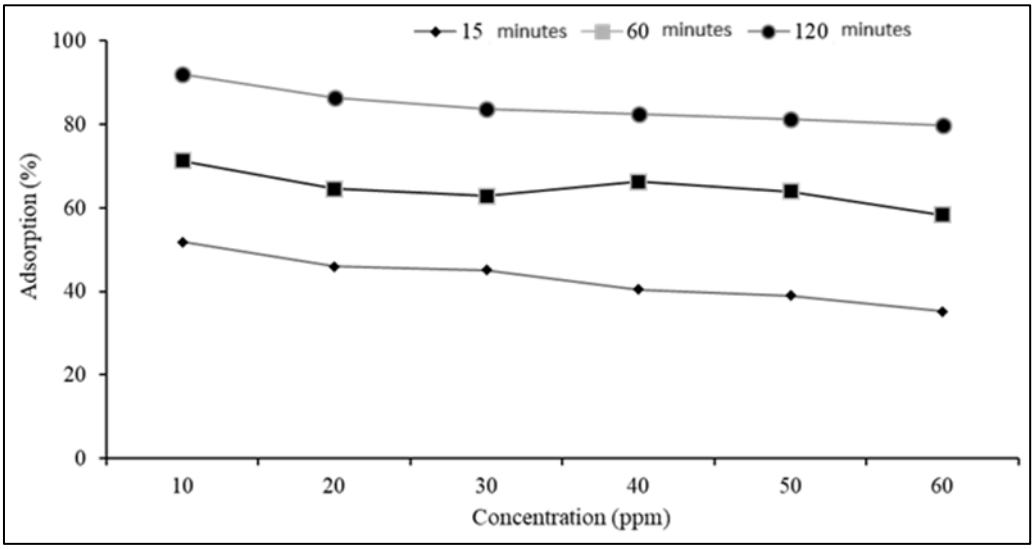

Figure 3 Effect of contact time on the adsorption of Cr^{6+} ions

Figure 3 demonstrates the significant impact of contact time on the adsorption efficiency of Cr^{6+} ions using 0.6 g of activated carbon at pH 2. The study explored initial concentrations of Cr^{6+} ranging from 10 to 60 ppm and evaluated adsorption at three time intervals: 15, 60, and 120 minutes. A clear trend was observed wherein the removal efficiency improved markedly with increased contact time. For instance, at a Cr^{6+} concentration of 10 ppm, the removal efficiency increased from approximately 53% at 15 minutes to around 80% at 120 minutes. This enhancement can be attributed to the gradual occupation of adsorption sites as the process progresses. Notably, the adsorption capacity after 120 minutes consistently surpassed that at 60 and 15 minutes across all tested concentrations. However, a plateau in the removal efficiency at 120 minutes suggests that equilibrium was achieved, indicating saturation of most accessible adsorption sites on the activated carbon. Beyond this point, further increases in contact time yielded only marginal improvements in adsorption, implying limited availability of unoccupied or energetically favorable binding sites. Mechanistically, the rapid uptake in the initial phase (15–60 minutes) is likely driven by the high number of available active sites and a steep concentration gradient, facilitating fast mass transfer. As the sites become progressively filled, steric hindrance, repulsive electrostatic forces, and diffusion limitations contribute to a slower adsorption rate. These findings have practical implications for the design of batch adsorption systems, particularly in wastewater treatment. They indicate that a contact time of approximately 120 minutes is optimal to maximize Cr^{6+} removal under the specified conditions, enabling effective process planning and resource utilization.

3.5 Surface morphology of activated carbon derived from brown seaweed waste

The surface morphology of the activated carbon synthesized from brown seaweed residue was examined using Scanning Electron Microscopy (SEM), as shown in Figure 4. The micrograph reveals a rough, fractured structure with a highly porous surface, characterized by numerous irregular cracks, flakes, and cavities. This morphological pattern suggests a well-developed network of micro- and mesopores, which is typical of biochar materials derived from lignocellulosic biomass after pyrolysis and chemical activation.

The porous texture significantly increases the surface area available for adsorption, thereby enhancing the material's capacity to capture Cr^{6+} ions from aqueous solutions. The fractured layers and open pore channels facilitate both surface adsorption and internal diffusion of metal ions, which is crucial for achieving high removal efficiency. The formation of such structures is likely attributed to the decomposition of organic matter and release of volatile compounds during thermal treatment, resulting in the expansion and collapse of cellular walls in the seaweed matrix.

These morphological features not only confirm the successful activation of the seaweed biomass but also directly correlate with the high adsorption performance observed in batch experiments. The abundance of accessible adsorption sites, coupled with the interconnected pore network, makes this material a promising candidate for low-cost, eco-friendly water treatment applications, especially in the removal of toxic heavy metals like Cr^{6+}.

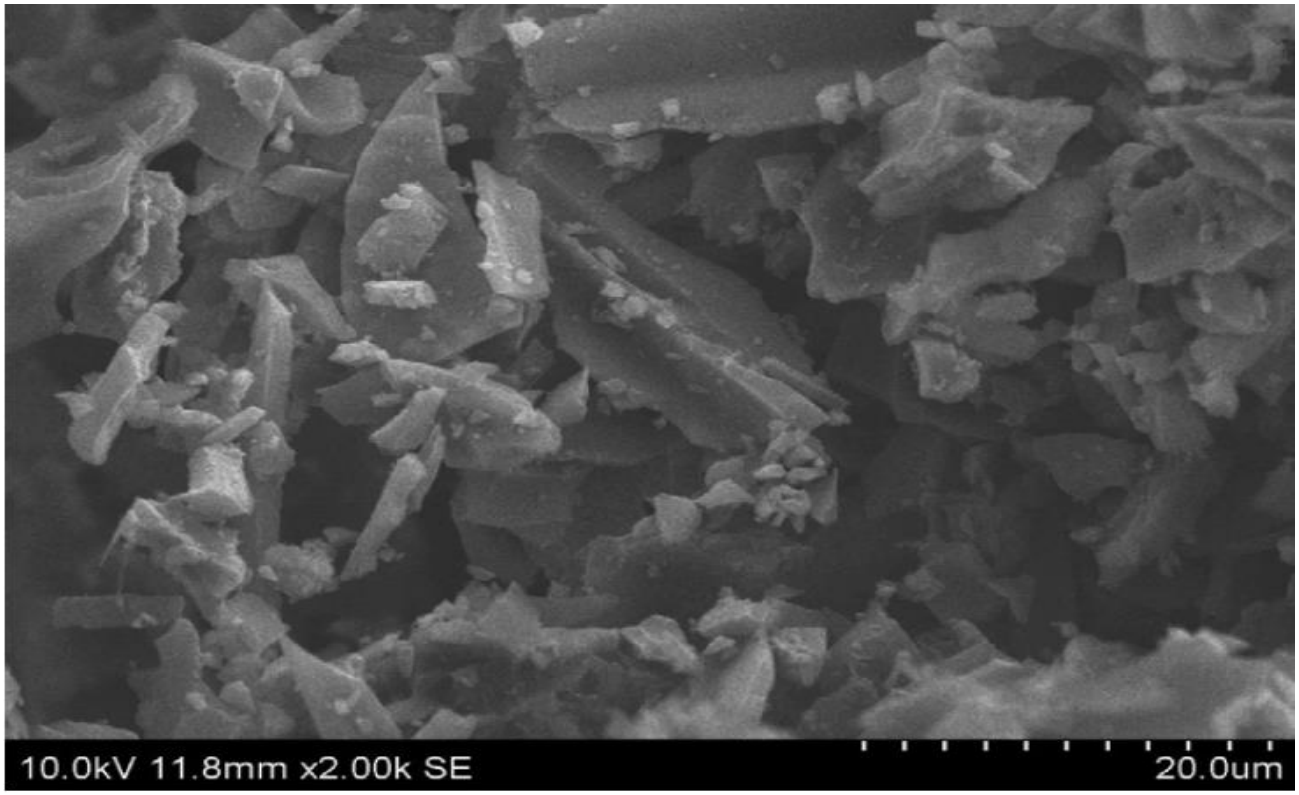

Figure 4 Activated carbon from brown algae waste under scanning electron microscope

3.6 Preparation processing of activated carbon from brown seaweed waste

The preparation of activated carbon (AC) from brown seaweed waste was conducted following a multistep protocol involving size separation, chemical pretreatment with zinc chloride, drying, carbonization, and activation, as outlined below.

3.6.1 Raw material preparation and fractionation

Dried brown seaweed was first cleaned and milled to remove impurities and reduce particle size. The milled seaweed was then sieved into two size fractions:

- MA fraction: particles smaller than 0.5 mm (150 g)
- MB fraction: particles larger than 0.5 mm (160 g)

3.6.2 Impregnation with $ZnCl_2$

Each fraction was subjected to chemical activation via impregnation with a 20% w/v solution of zinc chloride ($ZnCl_2$):

- MA: mixed with 1,500 mL of 20% $ZnCl_2$
- MB: mixed with 1,600 mL of 20% $ZnCl_2$

The mixtures were allowed to soak for 24 hours to ensure sufficient $ZnCl_2$ penetration. Subsequently, the impregnated biomass was dried in a hot-air oven at 100°C for 24 hours to remove moisture and partially fix the activating agent.

3.6.3 Carbonization and activation

The dried, impregnated seaweed samples were subjected to carbonization under the following conditions:

- Furnace temperature: 600°C
- Heating duration: 30 minutes
- Atmosphere: limited oxygen (semi-closed system to avoid complete combustion)

After carbonization, the resultant char was washed thoroughly with hot distilled water until the filtrate reached neutral pH, indicating the removal of residual $ZnCl_2$ and soluble by-products. The char was then oven-dried at 100°C and weighed to determine yield.

3.6.4 Yield of activated carbon

- From 150 g of MA fraction, 36 g of activated carbon was obtained (yield: 24%).
- From 160 g of MB fraction, 34 g of activated carbon was obtained (yield: 21%).

These results suggest that smaller particle size (MA < 0.5 mm) led to slightly higher carbonization yield under the same activation conditions, potentially due to improved $ZnCl_2$ penetration and more efficient heat transfer during pyrolysis.

4 Conclusion

This research demonstrated the successful preparation of activated carbon from brown seaweed waste using $ZnCl_2$ chemical activation and controlled thermal treatment. The process produced activated carbons with good yields (24% for MA and 21% for MB fractions) and a well-developed porous structure, as confirmed by SEM imaging. The materials exhibited high adsorption efficiency for Cr^{6+} ions, with performance strongly influenced by pH, initial metal ion concentration, and contact time.

These findings indicate that brown seaweed, a renewable marine biomass, can be effectively converted into value-added adsorbents for heavy metal removal from wastewater. The approach contributes to sustainable waste management and supports circular economy principles while offering a low-cost alternative to commercial activated carbons for environmental remediation.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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